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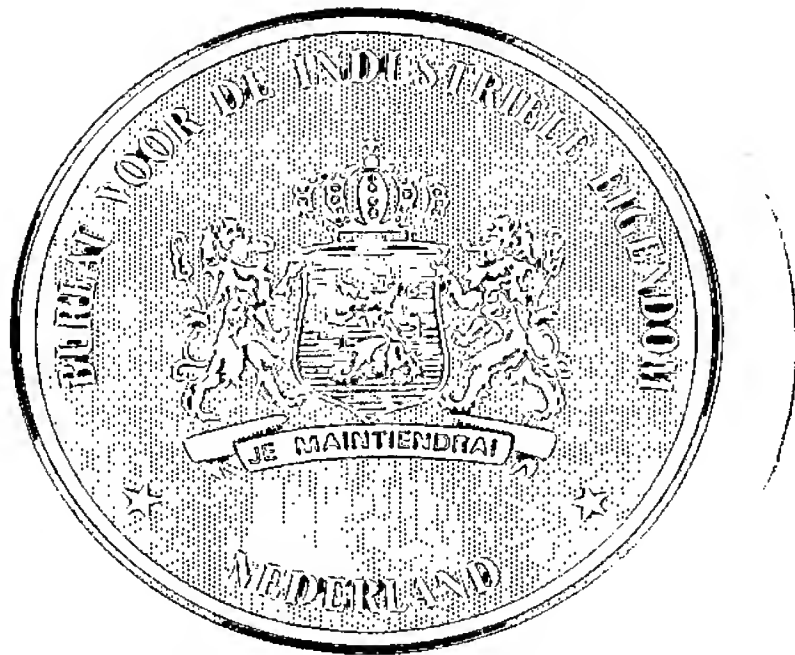


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This is to declare that in the Netherlands on January 19, 2004 under No. PCT/NL2004/00042,
in the name of:

DSM IP ASSETS B.V.

in Heerlen, the Netherlands (NL) and

Godfried Johannes Franciscus NIEMARK

in Amstenrade, the Netherlands (NL) and

Marianne SARKISSOVA

in Geleen, the Netherlands (NL)

an international patent application was filed for:

"Thermoplastic elastomer composition",

and that the documents attached hereto correspond with the originally filed documents.

Rijswijk, February 15, 2005

In the name of the president of the Netherlands Industrial Property Office



Mrs. D.L.M. Brouwer

THERMOPLASTIC ELASTOMER COMPOSITION

5 The present invention relates to a thermoplastic elastomer composition. The invention further relates to articles comprising the thermoplastic elastomer composition. The invention also relates to the use of the thermoplastic elastomer composition.

10 Thermoplastic elastomer compositions are known from "Handbook of Thermoplastic Elastomers, chapter 3, Van Nostrand Reinhold, New York (19**). The described thermoplastic elastomer composition comprises a blend of a thermoplastic polyolefinic polymer, an elastomer and oil.

15 Although the known thermoplastic elastomer compositions often possess desirable properties, the compositions seem not always preferred in for example medical, food or auto interior applications because of the migration of oil or other low molecular components present in the thermoplastic elastomer composition. Migration of oil is a problem, which is known in for example automotive interior applications as "blooming of oil". There is a need to reduce the migration of oil also within, for example packed food products and, particularly food products comprising fat
20 otherwise the food will be unusable for consumption. There is a need for thermoplastic elastomer compositions having low oil migration properties.

 The object of the present invention is to provide a thermoplastic elastomer composition with low oil migration properties.

25 This object is achieved by a thermoplastic elastomer composition comprising

- (a) 5-95 weight % of an oil free elastomer
- (b) 95-5 weight% of a polyolefin composition comprising from 20 to 50 parts by weight of a crystalline polyolefinic polymer and 50 to 80 parts by weight of an elastomeric olefinic copolymer whereby the total parts by weight is 100.

30 Surprisingly, it has been found that a significant reduction of migration of oil is achieved when the thermoplastic elastomer composition comprises the components (a) and (b). Another advantage is that good physical properties are retained especially relating to the hardness and the elasticity of the thermoplastic elastomer composition. Yet another advantage is that the thermoplastic elastomer
35 compositions are processable using different processing techniques.

 The oil free elastomers (a) according to the present

invention are elastomers which have not been extended by appropriate amounts of oil also called non oil-extended elastomers.. Examples of the non oil-extended elastomers are ethylene-propylene copolymers, hereinafter called EPM, ethylene-propylene-diene terpolymers, hereinafter called EPDM, natural rubbers, styrene-butadiene rubber (SBR), nitrile-butadiene rubbers (NBR), polyisoprene, butyl rubber or halogenated butyl rubber. Also other food approved rubbers can be used as non-oil-extended elastomer. Preferably EPM or EPDM is used as non oil-extended elastomer. The EPDM preferably contains 50-90 parts by weight ethylene monomer units, 48-30 parts by weight monomer units originating from an alpha-olefin and 1-12 parts by weight monomer units originating from a non-conjugated diene. As alpha-olefin use is preferably made of propylene. As non-conjugated diene use is preferably made of dicyclopentadiene (DCPD), 5-ethylidene-2- norbornene (ENB) or vinylnorbornene (VNB) or mixtures thereof. More preferably a mixture of EPM and EPDM is used as oil free elastomer elastomer. In case that a mixture of EPM and EPDM is used, the weight ratio EPM/EPDM may vary between 10/90 and 90/10. The amount of oil free elastomer varies between 5-95 weight %, preferably between 25-75 weight % of the thermoplastic elastomer composition. The oil free elastomer may however also be replaced by other polymers as far as the do not comprise oil. An example of other polymers is a styrene based polymer for example SBS, SEBS or SIPS.

The polyolefin composition (b) comprises from 20 to 50 parts by weight of a crystalline polyolefinic polymer and 50 to 80 parts by weight of an elastomeric olefinic copolymer whereby the total parts by weight is 100. More preferably, the polyolefin compositions comprise between 20 and 40 parts by weight of a crystalline polyolefinic polymer and between 60 and 80 parts by weight of an elastomeric olefinic copolymer whereby the total parts by weight is 100. Most preferably between 30 and 40 parts of a crystalline polyolefinic polymer and between 60 and 70 parts of an elastomeric olefinic copolymer whereby the total parts by weight is 100.

Preferably the polyolefin composition comprises 20 to 50 parts by weight of a crystalline polyolefinic polymer and from 50 to 80 parts by weight of an elastomeric olefinic copolymer with olefins $\text{CH}_2=\text{CHR}$, in which R is alkyl having 1 to 10 carbon atoms, and, if appropriate, containing minor proportions of units derived from a polyene.

The crystalline polyolefinic polymer is preferably selected from polypropylene homopolymer and propylene copolymers containing 0.5 to 15 mol % of ethylene and/or an .alpha.-olefin having 4 to 10 carbon atoms, the said propylene polymer for example has a molecular weight distribution (MWD) greater than 3.5.

The propylene homo polymer preferably has an isotactic index, determined by measurement of the solubility in xylene, greater than 85 and more preferably greater than 90. It is preferable for the propylene polymer to have an MWD greater than 5 and generally between 5 and 50. The melt index (ASTM 1238 condition "L") of the propylene polymer is generally between 0.1 and 50 g/10 minutes. Preferably the melt index is between 0.1 and 30. The propylene copolymer for example comprises from 2 to 10 mol % of an alpha-olefin other than propylene. Preferably, the alpha-olefin is selected from the group comprising ethylene, 1-butene, 1-hexene, 1-octene and 4-methyl-1-pentene. Among these, ethylene and 1-butene are particularly preferred.

The propylene polymers of the present invention can be prepared according to known technologies by polymerization of propylene, if appropriate in the presence of ethylene or other alpha.-olefins, in the presence of conventional catalysts of the Ziegler/Natta type comprising the product of the reaction between an aluminium alkyl and a solid component comprising a transition metal supported on $MgCl_2$ in an active form. Suitable methods for preparing the propylene polymers are described, for example, in EP-A-395083, EP-A-553805 and EP-A-553806, the description of which, relating to the method of preparation and to the characteristics of the products, is incorporated herein by reference.

The elastomeric olefinic copolymer is preferably selected from the copolymers of ethylene with alpha.-olefins $CH_2=CHR$ in which R is alkyl having 1 to 6 carbon atoms. More preferably, the alpha.-olefin is propylene or butene. In said copolymer, the content by weight of units derived from ethylene is preferably between 40 and 70%, more preferably between 50 and 70% most preferable between 60 and 70%. The content by weight of units derived from alpha.-olefins is preferably between 30 and 60%, more preferably between 30 and 50%, most preferably between 30 and 40%.

Particularly preferred are the copolymers, which contain 0.1 to 20% by weight, preferably 1 to 10%, of units derived from a polyene. Such a polyene can be selected from the group comprising trans-1,4-hexadiene, cis-1,4-hexadiene, 6-methyl-1,5-heptadiene, 3,7-dimethyl-1,6-octadiene and 11-methyl-1,10-dodecadiene; monocyclic diolefins such as, for example, cis-1,5-cyclooctadiene and 5-methyl-1,5-cyclooctadiene; bicyclic diolefins such as, for example, 4,5,8,9-tetrahydroindene and 6- and/or 7-methyl-4,5,8,9-tetrahydroindene; alkenyl- or alkylidene-norbornenes such as, for example, 5-ethylidene-2-norbornene, 5-isopropylidene-2-norbornene and exo-5-isopropenyl-2-norbornene; polycyclic diolefins such as, for example, dicyclopentadiene, tricyclo[6.2.1.0^{2,7}]4,9-undecadiene and the 4-methyl derivative thereof, 1,4-hexadiene,

isoprene, 1,3-butadiene, 1,5-hexadiene, 1,6-heptadiene and so on. Among these, 5-ethylidene-2-norbornene is particularly preferred. The elastomeric ethylene copolymer preferably has a low crystallinity. Preferably, the elastomeric ethylene copolymer used in the present invention has an MWD of lower than 3, generally of between 2 and 3.

5 The elastomer olefinic copolymer can advantageously be prepared by polymerizing mixtures of ethylene, alpha-olefin and, if appropriate, polyene in the presence of a catalytic system comprising a metallocene compound and an alumoxane.

10 The polyolefin compositions (b) can be prepared by using known methodologies such as mechanical mixing of the two components by means of internal mixers of the Banbury type, having a high homogenizing power. Alternatively, the said compositions can advantageously be obtained directly in the reactor by means of sequential polymerization. The compositions obtained according to this technique in fact show better elastomeric properties than those of the compositions obtained by
15 simple mechanical mixing. The above-mentioned polyolefine compositions (b) are preferably prepared by the process described in WO-A-9635751. These polyolefine compositions, also known as reactor TPO's, are commercially available for example under the tradename Hifax®.

20 The polyolefin compositions (b) may however be replaced by other polymers for example by amorphous polypropylene or ethylene copolymers known in the prior art as Plastomers.

 The thermoplastic elastomer composition comprising (a) and (b) preferably comprises 0,02-10 weight % of a crosslinking agent optionally with co-agent whereby the total weight of the thermoplastic elastomer composition is 100.

25 The crosslinking agents which can be used are those commonly known in the art, such as sulfur, sulfurous compounds, metal oxides, maleimides, phenol resins or peroxides. These vulcanization systems are known from the state of the art and are described in US-A-5100947. It is also possible to use silane compounds as vulcanization agent, examples are hydrosilane or vinylalkoxysilane. Possible is to
30 subject the elastomer to free radical grafting with a silane which has at least one olefinic double bond and one to three alkoxy groups bonded directly to the silicon. The grafted elastomer is crosslinked under influence of H₂O or condensation reaction. This vulcanization system is known from the state of the art and described in EP-A-510559. Preferably organic peroxides, phenolic resins or hydrosilanes are used as crosslinking
35 agent.

 Examples organic peroxides are dicumyl peroxide, di-tert-butylperoxide, 2,5-dimethyl-(2,5-di-tert-butylperoxy)hexane, 1,3 -bis(tert-

butylperoxyisopropyl)benzene, 1,1-bis(tert-butylperoxy)-2,3,5-trimethylcyclohexane, benzoyl peroxide, 2,4-dichlorobenzoyl peroxide, tert-butyl peroxybenzoate, tert-butyl peroxyisopropylcarbonate, diacetyl peroxide, lauroyl peroxide, tert-butyl cumyl peroxide.

5 The amount of crosslinking agent is preferably between 0,02-10% by weight and more preferably between 0,4-5% by weight relative to the total weight of the thermoplastic elastomer composition.

A co-agent may optionally be used during the vulcanization. Examples of suitable co-agents are polybutylene, liquid 1,2-polybutadiene, isoprene, butadiene/isoprene mixtures, divinyl benzene, sulphur, p-quinondioxime, nitrobenzene, diphenylguanidine, triarylcyaurate, trimethylolpropane-N,N-m-phenylenedimaleimide, ethyleneglycol dimethacrylate, polyethylene dimethacrylate, trimethylolpropane trimethacrylate, arylmethacrylate, vinylbutylate and vinylstearate. The amount of co-agent is preferably between 0-2.00% by weight relative to the total weight of the thermoplastic elastomer composition.

In case of vulcanisation, dynamically vulcanisation is preferred. When working according to this technique, the composition of the invention is subjected to kneading or to other shear forces in the presence of crosslinking agents and, if appropriate, co-agents at temperatures between for example 140 and 300° C., preferably at temperatures between 240 and 300° C or more preferably at temperatures higher than the melting point of the crystalline phase.

The degree of vulcanization of the elastomer composition can be expressed in terms of gel content. Gel content is the ratio of the amount of non-soluble elastomer and the total amount of elastomer (in weight) of a specimen soaked in an organic solvent for the elastomer. The method is described in US-A-5100947. In general terms a specimen is soaked for 48 hours in an organic solvent for the elastomer at room temperature. After weighing of both the specimen and the residue the amount of non-soluble elastomer and total elastomer are calculated, based on knowledge of the relative amounts of all components in the thermoplastic elastomer composition. The elastomer composition is at least partially vulcanized.

The thermoplastic elastomer composition according to the present invention may also comprise for example reinforcing and non-reinforcing fillers, plasticizers, antioxidants, stabilizers, antistatic agents, waxes, foaming agents, lubricants, pigments, flame retardants and other known agents described in for example the Rubber World Magazine Blue Book. Examples of fillers that may be used are calcium carbonate, clay, silica, talc, titanium dioxide, and carbon. Examples of lubricants are natural products like fatty acids, wool grease, glue, rosin or modified

natural products like factice. Of course a minimum amount of oil can be used as lubricant in the thermoplastic elastomer composition according to the present invention as long as low oil migration properties are obtained. Another additive that may optionally be used in the thermoplastic elastomer composition is a Lewis base for example a metal oxide, a metal hydroxide, a metal carbonate or hydrotalcite.

The thermoplastic elastomer composition (a), (b) of the present invention may additionally comprise a styrene-based polymer. Examples of styrene-based polymers are styrene-isobutylene-styrene (SIBS), styrene-isobutylene (SIB), styrene-ethylene-butylene-styrene polymers (SEBS) or styrene-butadiene-styrene polymers abbreviated as (SBS). Preferably SBS or SEBS is used as styrene based polymer. The amount of the styrene-based polymer may for example vary between 2-50 weight %, preferably between 5-40 weight %, more preferably between 5-20 weight % whereby the total weight of the thermoplastic elastomer composition is 100.

The thermoplastic elastomer composition is for example prepared in conventional mixing equipment for example a Banbury mixer, a Brabender mixer, a continuous mixer for example a single screw extruder, a twin screw extruder and the like. Preferably the thermoplastic elastomer composition is prepared in a twin screw extruder. In case of vulcanising, the crosslinking agent and optionally co-agent, may be fed in whole or in part at a place downstream in the extruder. In case that a styrene-based polymer is present in the thermoplastic elastomer composition the crosslinking agent may be fed before or after the styrene-based polymer. Preferably it is fed before injecting the styrene-based polymer. The styrene-based polymer may also be fed at the entry port of the extruder together with the oil free elastomer (a) and the polyolefin composition (b). The thermoplastic elastomer composition may also be prepared in a two-steps process by feeding the oil free elastomer (a), the polyolefin composition (b) and usual additives to a batch kneader in order to mix the composition wereafter granules may be produced. In case of vulcanising, the granules may for example be fed to a twin-screw extruder. The crosslinking agent and optionally the styrene-based polymer may be fed in whole or in part at a place downstream in the extruder. The styrene polymer may however also be fed to the batch kneader or be fed to the entry port of the twin screw extruder.

The thermoplastic elastomer composition according to the present invention surprisingly has a low hardness which the polymers processable using different techniques. The hardness is for example below 70 Shore A, preferably below 65 Shore A.

The thermoplastic elastomer composition according to the present invention can be used for the manufacturing of articles in all processes including for

example by extrusion, molding for example injection molding or blow molding or thermoforming. Examples of articles are writing utensils, seals for various containers, preservation jars, sealing gaskets for food savers, caps and closure seals and seals for various containers.

5 The thermoplastic elastomer composition according to the present invention may be used in food packaging, industrial applications, consumer applications, medical applications and seals auto interior parts and low fogging applications as for example airbags and dashboards.

10 The following examples are given for illustrative purposes and do not limit the invention itself.

 The different properties of the thermoplastic elastomer composition were analysed by using the following test methods:

- Hardness according to ASTM D-2240
- Apparant Shear Viscosity at 206 1/s, at 220C according to ASTM D-3835
- 15 • Oil migration according to EC 2002-72

 Regarding the oil migration properties the amount of extractables the thermoplastic elastomer composition are determined according to the below described procedure:

20 95 % Ethanol was used as extraction liquid and prepared by addition of 950 ml ethanol (Lichrosolv, >99.9 %) to 50 ml demi-water and subsequent mixing.

Before use all glassware was washed extensively with extraction liquid.

- Samples were cut in pieces.
- Before extraction samples were cleaned with tissue and ethanol to remove dust and other contaminants.
- 25 • 100 ml Schott flasks were extensively washed with extraction liquid and dried.
- Approx. 1 dm² (1 dm² = 12.68 g) of the sample was put in Schott flasks.
- Approx. 100 ml extraction liquid per dm² was added to the flask.
- The samples were extracted for 240h hours at 40°C.
- The extraction liquid was evaporated to dryness in an aluminum dish at 50 °C. The
- 30 flasks were flushed with 3 * 20 ml extraction liquid, which was also evaporated.
- Before use aluminum dishes were extensively washed with extraction liquid, dried and weighed.
- The residues were determined by weighting.
- Experiment was conducted in 7-fold.
- 35 • A blank (everything without sample) was also conducted. Results were corrected for the blank value.

Example 1

21,53 weight % of EPM (ML (1+4) 100 C 51) is mixed with 75,35 weight% Hifax 7334 XEP® and 0,43 weight % of additives on a 40mm co-rotating twin screw extruder at a melt temperature of 260°C. Downstream in the extruder 2,69 weight % of Trigonox 101E30® is injected. The properties and results of this composition are shown in table 1.

Example 2

14,40 weight% of EPM (ML (1+4) 100 C 51), 14,40 weight% of EPDM (ML (1+4) 100 C 55), 67,21 weight % of Hifax 7334 XEP® and 0,38 weight % of additives are mixed on a 40mm co-rotating twin screw extruder at a melt temperature of 260°C. Downstream in the extruder 3,60 weight % of Trigonox 101E30® is injected. The properties and results of this composition are shown in table

Example 3

17,91 weight% of EPM (ML (1+4) 100 C 51), 8,96 weight% of EPDM (ML (1+4) 100 C 55), 62,70 weight % of Hifax 7334 XEP®, 0,35 weight % of additives and 6,72 weight% of SEBS are mixed on a 40mm co-rotating twin screw extruder at a melt temperature of 260°C. Downstream in the extruder 3,36 weight % of Trigonox 101E30® is injected. The properties and results of this composition are shown in table1.

Example 4

14,40 weight% of EPM (ML (1+4) 100 C 51), 14,40 weight% of EPDM (ML (1+4) 100 C 55), 70,81 weight % of Hifax 7334 XEP® and 0,38 weight % of additives are mixed on a 40mm co-rotating twin screw extruder at a melt temperature of 260°C. The properties and results of this composition are shown in table 1.

Example 5

14,40 weight% of EPM (ML (1+4) 100 C 51), 14,40 weight% of EPDM (ML (1+4) 100 C 55), 62,21 weight % of Hifax 7334 XEP® and 0,35 weight % of additives are mixed on a 40mm co-rotating twin screw extruder at a melt temperature of 260°C. Downstream in the extruder 3,36 weight % of Trigonox 101E30® and 5 weight% of Ricon 154D® is injected. The properties and results of this composition are shown in table 1.

Comparative experiment A

62,32 weight% of oil extended EPDM comprising 100 phr of oil, 14,08 weight % of polypropylene and 5,87 weight % talc are mixed on a 40mm co-rotating screw extruder.

- 5 Downstream 0,94 weight %. Trigenox 101 E 30® and 0,32 weight % co-agent are injected. The properties and results are shown in table 2.

Table 1

	Example 1	Example 2	Example 3	Example 4	Example 5
	weight %	weight %	weight %	weight %	weight %
EPM (ML (1+4) 100 °C 51)	21.53	14.40	17.91	14.40	14.40
EPDM (ML (1+4) 100 °C 55)		14.40	8.96	14.40	14.40
Hifax 7334 XEP®	75.35	67.21	62.70	70.81	62.21
SEBS			6.72		
Trigenox 101E30®	2.69	3.60	3.36		3.36
Ricon 154D®					5.00
Additives	0.43	0.38	0.35	0.38	0.35
Hardness Shore A	65	60	66	68	61
CS at 23 °C for 72h	69	83	64	94	38
Viscosity 220°C(Pa.s)	843	541	480	365	320
Migration (mg/dm ²)	23	18	26	15	11

Table 2

Comparative exp. A	Weight %
EPDM, oil extended	60.32
Polypropylene	14.18
Talc	5.87
Trigonox 101 ^E 30	0.94
Co-agent	0.32
Additive	0.39
Oil	18.09
Hardness shore A	62
CS at 23° C for 72h	24
Vicinity	180
Migration	292

From table 1, example 1 it is clear that low oil migration properties are achieved by vulcanised thermoplastic elastomer composition comprising an oil free elastomer and a polyolefine composition.

From example 2 it is clear that a vulcanised thermoplastic elastomer composition comprising 2 different oil free elastomers and a polyolefine composition results in even lower oil migration properties. As shown in example 3 the addition of SEBS highers the oil migration properties negligible. From example 4 it is clear that also no vulcanized thermoplastic elastomer compositions show low oil migration properties. As can be derived from example 5 the oil migration properties are at a very low level in case that the thermoplastic elastomer composition is crosslinked in the presence of a co-agent.

From table 2, it is clear that a vulcanized thermoplastic elastomer comprising the above components results in bad oil migration properties.

CLAIMS

1. A thermoplastic elastomer composition comprising:
(a) 5-95 weight % of an oil free elastomer
5 (b) 95-5 weight% of a polyolefin composition comprising from 20 to 50 parts by weight of a crystalline polyolefinic polymer and 50 to 80 parts by weight of an elastomeric olefinic copolymer whereby the total parts by weight is 100.
2. A thermoplastic elastomer composition according to claim 1 comprising 0,02-10 weight % of a crosslinking agent optionally with co-agent whereby the total weight of (a), (b) and the crosslinking agent is 100.
3. A thermoplastic elastomer composition according to claim 2 characterised in that the crosslinking agent is chosen from a peroxide, hydrosilane or phenolic resins.
4. A thermoplastic elastomer composition according to any one of the claims 1-3 characterised in that it the oil free elastomer (a) is chosen from EPM, EPDM or a mixture of EPM and EPDM.
5. A thermoplastic elastomer composition according to any one of the claims 1-4 characterised in that the polyolefin composition (b) comprises a polypropylene homopolymer or propylene copolymer as crystalline polyolefinic polymer and a ethylene copolymer with olefins having the formula $CH_2=CHR$ in which R is an alkyl having 1-10 carbon atoms as elastomeric olefinic copolymer.
6. A thermoplastic elastomer composition according to any one of the claims 1-5 characterised in that the composition comprises a styrene based polymer whereby the total weight of the thermoplastic elastomer composition is 100.
7. A thermoplastic elastomer composition according to any one of the claims 1-6 characterised in that the composition comprises 2-50 weight % of a styrene based polymer whereby the total weight of the thermoplastic elastomer composition is 100.
8. A thermoplastic elastomer composition according to any one of the claims 6-7 characterised in that the styrene-based polymer is chosen from SBS, SEBS or SIPS.
9. A thermoplastic elastomer composition according to any one of the claims 1-8 characterised in that the co-agent is chosen from polybutylene, isoprene or mixtures of isoprene and butadiene.
10. Articles comprising the thermoplastic elastomer composition according to any one of claims 1-9.

11. Use of the thermoplastic elastomer composition according to claims 1-9 in food packaging, industrial applications, consumer applications, medical applications, seals, auto interior parts and low fogging applications as for example airbags and dashboards.
- 5

ABSTRACT

- A thermoplastic elastomer composition comprising 5-95 weight % of an oil free elastomer, 95-5 weight% of a polyolefin composition comprising from
- 5 20 to 50 parts by weight of a crystalline polyolefinic polymer and 50 to 80 parts by weight of an elastomeric olefinic copolymer whereby the total parts by weight is 100. The thermoplastic elastomer composition may comprise 0,02-10 weight % of a crosslinking agent optionally with co-agent whereby the total weight of (a), (b) and the crosslinking agent is chosen from a peroxide, hydrosilane or phenolic resins.
- 10 The present invention further relates to articles comprising the thermoplastic elastomer composition and to the use of the thermoplastic elastomer composition in food packaging, industrial application, consumer applications, medical applications, seals, auto interior parts and low fogging applications as for example airbags and dashboards.